

# The Effect of Leveling Coatings on the Atomic Oxygen Durability of Solar Concentrator Surfaces

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# **THE EFFECT OF LEVELING COATINGS ON THE ATOMIC OXYGEN DURABILITY OF SOLAR CONCENTRATOR SURFACES**

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## **SUMMARY**

Space power systems for Space Station Freedom will be exposed to the harsh environment of low Earth orbit (LEO). Neutral atomic oxygen is the major constituent in LEO and has the potential of severely reducing the efficiency of solar dynamic power systems through degradation of the concentrator surfaces. Several transparent dielectric thin films have been found to provide atomic oxygen protection, but atomic oxygen undercutting at inherent defect sites is still a threat to solar dynamic power system survivability. Leveling coatings smooth microscopically rough surfaces, thus eliminating potential defect sites prone to oxidation attack on concentrator surfaces. This paper investigates the ability of leveling coatings to improve the atomic oxygen durability of concentrator surfaces. The application of an EPO-TEK<sup>R</sup> 377 epoxy leveling coating on a graphite epoxy substrate resulted in an increase in solar specular reflectance, a decrease in the atomic oxygen defect density by an order of magnitude and a corresponding order of magnitude decrease in the percent loss of specular reflectance during atomic oxygen plasma ashing.

## **INTRODUCTION**

Electrical power of 75 kilowatts for the initial phase of Space Station Freedom (SSF) will be generated by traditional photovoltaic (PV) power sources. Power will be increased during the growth phase ( $\approx 4$  years after the initial phase begins) by the addition of two solar dynamic (SD) power modules<sup>1</sup>. Each SD power module will be capable of generating 50 kW of available power.

In a solar dynamic power system, a solar concentrator collects and focuses the sun's light into the aperture of a heat engine such as a closed Brayton cycle system<sup>1</sup>. In such a system a gaseous working fluid is heated by solar energy in the heat receiver, and converted to electricity by a power conversion unit<sup>1</sup>. Solar dynamic systems have the potential for significantly higher solar-to-electric power efficiency than

PV systems, with about 60% smaller solar collection area for a given power output<sup>1</sup>. This can result in a substantial savings over the life of SSF due to lower aerodynamic drag and lower reboost requirements<sup>1</sup>. Figure 1 shows the current solar concentrator configuration for SSF solar dynamic power modules<sup>2,3</sup>. The solar concentrator is comprised of 19 hexagonal elements, each comprised of 24 spherically contoured triangular facets measuring 1 meter per side. The mirror facets are composed of two sheets of graphite epoxy bonded to an Al honey-comb core. Reflective and protective thin film coatings are deposited onto the graphite epoxy face sheet<sup>4</sup>.

The efficiency of a SD system depends on the ability of the solar concentrator to accurately direct the maximum amount of the sun's incident rays into the heat receiver. Thus, the concentrator must have and maintain a high solar specular reflectance. Solar specular reflectance is defined as the percentage of incident solar energy reflected through an aperture of a given solid angle. In this case, the aperture is defined by the diameter and orientation of the heat receiver aperture and the distance from the concentrator to the receiver. Diffusely reflected radiation from the concentrator will not be adequately directed into the heat receiver, and thus is almost entirely wasted energy.

Low Earth orbital (LEO) environment, in which Space Station Freedom will operate, is comprised of many elements which can cause considerable damage to power system materials. Neutral atomic oxygen is the predominant species in LEO<sup>5</sup> (see figure 2), and is a threat to the survivability of both PV and SD systems<sup>6,7</sup>. Other harmful LEO elements include UV radiation, thermal cycling, micrometeoroid and debris impacts and synergistic effects of these elements<sup>2</sup>.

Survivability of the concentrator reflective media in the harsh LEO environment is essential for power generation on a SD system. Several transparent dielectric thin films have been identified as atomic oxygen protective films, including  $\text{MgF}_2$ ,  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$ <sup>4,6</sup>. Although metal oxides are found to be atomic oxygen protective, atomic oxygen undercutting at inherent defect sites is still a threat to SD survivability.

Undercutting of oxidizable substrate materials at defect sites in protective coatings, in which the damaged or oxidized area extends far beyond that of the original defect area, has been studied extensively at the NASA Lewis Research Center<sup>2,8,9,10,11,12</sup>. The potentially catastrophic result of atomic oxygen undercutting in LEO is receiving widespread recognition as a threat to spacecraft materials survivability.

Inherent atomic oxygen transparent defects (transparent being those in which atomic oxygen can penetrate) are formed during fabrication. Atomic oxygen transparent defects can also be introduced during handling, deployment, or from micrometeoroid and space debris impact. Although it will not be possible to affect the number of particle impacts occurring in space, we can greatly reduce the number of atomic oxygen transparent defects which occur during fabrication and handling. The control of defect density in protective coatings appears to be the key to long-term protection of solar concentrator surfaces in LEO<sup>8</sup>.

The SSF concentrator graphite epoxy substrate is fabricated by transfer casting. Both the mold release agent and the mold surface finish will partially determine the smoothness of the transfer cast composite face sheet of the concentrator<sup>2</sup>. Graphite epoxy print-through, caused by epoxy shrinkage during polymerization produces a macroscopic fiber pattern on the surface of the composite<sup>2</sup>. Both print-through and transfer cast surface roughness can be sites for atomic oxygen defects. The planned multi-layer reflective and protective thin films for the SSF concentrator surface will be deposited by electron beam evaporation. Evaporation is a line-of-sight deposition technique; microscopic and macroscopic roughness, as formed during transfer casting and from print-through, along with dust particles on the surface can be sites of discontinuity in the deposited films and thus potential atomic oxygen defect sites.

This study uses a RF plasma asher to simulate the LEO atomic oxygen environment<sup>12</sup>. Figure 3a illustrates atomic oxygen undercutting at a protective coating defect site on a graphite epoxy substrate. The shallow but very wide undercut region is representative of the shape of undercutting which occurs in an atomic oxygen plasma environment. The micrograph in figure 3b illustrates that atomic oxygen damage can occur to solar concentrator surfaces with a multi-layer protective system. This sample has the same reflective, protective, and substrate layers which are currently planned for SSF concentrator surfaces: a graphite epoxy substrate, an adhesion promoting layer of Cu, a Ag reflective layer, and two atomic oxygen protective layers, SiO<sub>2</sub> on Al<sub>2</sub>O<sub>3</sub>. The aluminum oxide also acts as an adhesion promotor between SiO<sub>2</sub> and Ag.

Leveling coatings have generally been applied as a means to increase the specular reflectance of optical surfaces. A lacquer leveling coating has been used successfully for producing very smooth surfaces for x-ray telescopes<sup>2</sup>. Sandia National Laboratories has applied sol-gel derived leveling coatings for increasing the solar specular reflectance of stainless steel substrates for solar mirrors<sup>13</sup>.

It is believed that the number of inherent atomic oxygen transparent defect sites can be greatly reduced through the application of a surface tension leveling coating. If a leveling coating is applied onto the graphite epoxy substrate prior to deposition of the reflective and protective layers, the rough undulations of the graphite epoxy can be smoothed, and electron beam deposition will produce continuous reflective and protective films. This in turn will decrease the number of atomic oxygen transparent defects. The proposed protection afforded by a leveling layer is shown schematically in figure 4. This paper addresses preliminary studies on the effectiveness of applying leveling coatings for decreasing the atomic oxygen defect density and thus increasing the LEO durability of solar concentrator surfaces.

## EXPERIMENTAL PROCEDURES

### Sample Fabrication

Mechanically polished (T-300/934) and unpolished (AS4/MY 720) graphite epoxy samples were prepared at NASA Lewis Research Center. The procedure for polishing was as follows: 600 grit emery paper, water rinse and final polish with 3 micron diamond paste. Both polished and unpolished graphite epoxy samples are similar in composition to the graphite epoxy for SSF solar concentrator substrates (polyacrylonitrile (PAN), preimpregnated epoxy with fibers (pregreg.) and unidirectional ply).

Several epoxies were evaluated as potential leveling coatings. Extra Fast Setting Epoxy (Hardman, Inc.), EPON<sup>R</sup> 815-Deta epoxy, EPON<sup>R</sup> 815-Tetra epoxy (Shell Chemical Co.), and EPO-TEK<sup>R</sup> 377 high temperature epoxy (Epoxy Technology, Inc.) were evaluated as potential leveling coatings. Surface tension leveled coatings were produced by preparing the epoxies as directed and pouring them onto the surface of the polished and unpolished graphite epoxy samples. The epoxy coated samples were then cured as directed. The EPON<sup>R</sup> 815 coated samples were put under vacuum before curing in an attempt to remove air bubbles.

Graphite epoxy samples which were polished, unpolished, and had leveling coatings, along with fused silica optical slides were coated with aluminum by electron beam evaporation using a CHA Industries Model 271 Four Pocket Electron Beam Gun equipped with an Eratron Model EB-8 Electron Beam Power Supply. Pressure during deposition was approximately  $1 \times 10^{-6}$  torr. Aluminum was chosen because it could serve as both the reflective and the protective layers for this study<sup>14</sup>. Aluminum film thickness was determined by profiling the fused silica optical slide using a Sloan Dektak II programmable diamond stylus profilometer.

### Atomic Oxygen Exposure

Samples were exposed to an atomic oxygen environment in a Structure Probe, Inc. Plasma Prep II asher. This asher generates a plasma by exciting ambient air with 100 W of continuous RF power at 13.56 MHz<sup>9</sup>. The operating pressure was approximately  $5.0 \times 10^{-2}$  torr. The effective atomic oxygen fluence was calculated based on mass loss data of 5 mil Kapton<sup>R</sup> H polyimide which was ashed along with the samples. An erosion yield of  $3 \times 10^{-24}$  cm<sup>3</sup>/atom for the Kapton<sup>R</sup> was assumed based on space flight data<sup>2,7</sup>.

### Reflectance Measurements

The reflectance measurements were obtained using a Perkin-Elmer Lambda 9 UV/VIS/NIR spectrophotometer operated with a 60 mm diameter barium sulfate coated integrating sphere. The specular reflectance was measured at an 8 degree angle from normal incidence, with an aperture solid angle of 0.096 steradians (230 x 320 mrad aperture). Integrated solar reflectances were obtained by measuring the

reflectance over the wavelength range 250-2500 nm and then using a NASA Lewis written program to convolute this spectrum into the air mass zero (AMO) solar spectrum over the same wavelength range<sup>10</sup>.

### **Scanning Electron Microscopy (SEM) & Defect Density Concentration**

Samples were coated with approximately 200Å of Au in preparation for examination on a Cambridge 200 SEM. Micrographs were taken at regular intervals along each sample for defect density counting. Both secondary electron (SE) and back scattered electron (BSE) images were obtained. Defect density concentrations were calculated from the SEM micrographs based on sites in which undercutting was apparent.

## **RESULTS AND DISCUSSION**

### **Solar Specular Reflectance**

Table 1 lists the solar specular reflectance of the evaluated samples before (pristine) and after ashing. Also included in this table is Al thickness, percent loss in solar specular reflectance due to ashing, and effective fluence for each sample. The specular reflectance of the EPON<sup>R</sup> 815-Deta, and two of the Extra Fast Setting Epoxy (EFSE) leveling coated samples (#1 and #2) were relatively high (0.793, 0.807 and 0.817, respectively). But, visual appearance of the EPON<sup>R</sup> 815 and EFSE leveling coated samples revealed various surface morphologies such as: bubbles, creased lines, and in the case of the EPON<sup>R</sup> 815 vacuum treated samples, many small swirled regions. After electron beam evaporation of Al onto the surfaces, some of the EFSE samples obtained a diffuse appearance which was attributed to sample heating during deposition. Ashing these samples produced a decrease in the solar specular reflectance from 6.1 to 57.5 percent. The exception was the EPON<sup>R</sup> 815-Tetra coated sample which showed an increase in the solar specular reflectance. This is attributed to a variation in the surface morphology across the surface; the exact region scanned was probably not the same before and after ashing. Since EPON<sup>R</sup> 815 and EFSE leveling coatings were not smooth, some became diffuse during evaporation, and had a large drop in specular reflectance with ashing; they were found to be inadequate as leveling coatings.

EPO-TEK<sup>R</sup> 377 high temperature epoxy produced a smooth leveling layer over the unpolished graphite epoxy substrates. The pristine integrated solar specular reflectance was increased from 0.098 for an unpolished graphite epoxy sample to 0.893 for an unpolished graphite epoxy with a leveling coating of EPO-TEK<sup>R</sup> 377 epoxy, an increase of  $\approx 800$  percent. This epoxy was not affected by heating during deposition or ashing. The loss in solar specular reflectance due to ashing ranged from 0.6% to 1.2% for a fluence of  $5.15 \times 10^{20}$  atoms/cm<sup>2</sup> ( $\approx 0.5$  yr SSF), and from 2.6% to 3.1% for a fluence of  $1.15 \times 10^{21}$  atoms/cm<sup>2</sup> ( $\approx 1.1$  yr SSF). These values are more than an order of magnitude lower than for unpolished graphite epoxy sample ashed with the same fluence, which resulted in losses of 35.7% and 44.3%,

respectively. It should be noted that although polishing the graphite epoxy can increase the solar specular reflectance, the application of a leveling coating can further increase the solar specular reflectance by an additional 54 percent (as found in this study). A comparison of the solar specular reflectance of Al-coated unpolished, polished, and leveling coated unpolished graphite epoxy samples are shown in figure 5.

### Defect Density Concentration

Many types of surface defects may be visible during SEM examination. Only the defects which are atomic oxygen transparent are a threat to the atomic oxygen durability of solar concentrator surfaces. Thus, only defects which undercut during ashing were counted. These defects can become visible during SEM examination in two ways, depending on the size of the undercut region and the materials involved. When nonconductive materials are examined, such as  $\text{SiO}_2$  on Kapton<sup>R</sup> polyimide, charging is usually evident in the normal secondary electron image. If conductive materials are examined, such as Al on graphite epoxy, charging does not always occur. By using backscattered electron imaging, undercutting of conductive materials can be observed as seen in figure 6. Backscattered images distinguish between light and heavy elements; light elements appear dark, heavy elements appear bright. Either oxidized or void regions would appear dark in the BSE image.

Both of the Al-coated unpolished graphite epoxy samples without leveling coatings did not readily show visible signs of undercutting in either SE or BSE imaging. Many of the fibers were directly exposed to the surface prior to Al deposition, and the fibers which were covered had a very thin layer of epoxy, so electron charging did not occur at the defect sites. In BSE imaging the underlying graphite fibers were visible as bright lines. This caused confusion in determining undercut areas in the BSE mode since it was difficult to distinguish between dark regions which were undercut and dark regions of epoxy. Defect density concentrations could not be obtained for these samples. The defect density concentration of the Al-coated polished graphite epoxy sample was obtained. Undercut areas which were not visible in the SE images were readily visible in the BSE images and used for atomic oxygen transparent defect counting. This sample, ashed to a fluence of  $8.99 \times 10^{20}$  atoms/cm<sup>2</sup>, resulted in a defect density concentration of 262,300 defects/cm<sup>2</sup>. Two of the Al-coated EPO-TEK<sup>R</sup> 377 coated samples were used for defect density counting: #3, ashed to a fluence of  $5.15 \times 10^{20}$  atoms/cm<sup>2</sup>, and #2, ashed to a fluence of  $1.15 \times 10^{21}$  atoms/cm<sup>2</sup>. These leveling coated samples resulted in defect density concentrations of 22,500 and 21,000 defects/cm<sup>2</sup>, respectively. Leveling coatings produced a decrease in the atomic oxygen defect density concentration by an order of magnitude (figure 7). In figure 8, SE and BSE micrographs of ashed uncoated and leveling coated samples show the increased atomic oxygen durability and smoothing effects provided by leveling coatings.



## CONCLUSION

Several types of epoxies were evaluated as potential leveling coatings for reducing the number of atomic oxygen transparent defects in protective coatings for solar concentrator surfaces. EPON<sup>R</sup> 815-Tetra, EPON<sup>R</sup> 815-Deta and Extra Fast Setting epoxies were found to be unsuitable as leveling coatings because they did not produce smooth coatings, some became diffuse during Al deposition and they displayed a large drop in specular reflectance with ashing. EPO-TEK<sup>R</sup> 377 high temperature epoxy produced a smooth surface tension leveling layer over an unpolished graphite epoxy substrate. An increase in the pristine solar specular reflectance of 811% was produced compared to an unpolished graphite epoxy coupon. Leveling coatings produced a decrease in the inherent atomic oxygen defect density concentration by an order of magnitude (262,300 to 22,000 defects/cm<sup>2</sup>), and a corresponding order of magnitude decrease in the percent loss of solar specular reflectance during ashing (35.7-44.3 percent to 0.6-3.1 percent).

The application of a leveling coating can increase the solar specular reflectance and the atomic oxygen durability of solar concentrator surfaces, increasing both the efficiency and the lifetime of solar dynamic power systems.

## ACKNOWLEDGEMENTS

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**TABLE I**  
**Solar Specular Reflectance of Pristine and Ashed Samples**

Sample Description Leveling Coating (Graphite Epoxy Substrate)		Substrate Polished	Solar Specular Reflt. <sup>a</sup> Integrated Values		% Loss in Specular Reflt. Due to Ashing
			Pristine	Ashed	
1	No Leveling Coat #1	No	0.098	0.063 <sup>1</sup>	35.7
2	No Leveling Coat #2	No	0.203	0.113 <sup>2</sup>	44.3
3	No Leveling Coat #2	Yes	0.580	0.566 <sup>3</sup>	2.4
4	EPO-TEK <sup>R</sup> 377 #1	No	0.860	0.855 <sup>1</sup>	0.6
5	EPO-TEK <sup>R</sup> 377 #2	No	0.883	0.860 <sup>2</sup>	2.6
6	EPO-TEK <sup>R</sup> 377 #3	No	0.893	0.882 <sup>1</sup>	1.2
7	EPO-TEK <sup>R</sup> 377 #4	No	0.892	0.864 <sup>2</sup>	3.1
8	EPON <sup>R</sup> 815-Tetra #1	No	0.392	0.415 <sup>1</sup>	-5.9
9	EPON <sup>R</sup> 815-Deta #1	No	0.793	0.337 <sup>1</sup>	57.5
10	Extra Fast Setting Epoxy #1	Yes	0.807	0.751 <sup>2</sup>	6.9
11	Extra Fast Setting Epoxy #2	Yes	0.817	0.767 <sup>2</sup>	6.1
12	Extra Fast Setting Epoxy #3	Yes	0.340	0.247 <sup>1</sup>	27.4
13	Extra Fast Setting Epoxy #4	Yes	0.356	0.300 <sup>1</sup>	15.7
14	Extra Fast Setting Epoxy #5	Yes	0.373	0.342 <sup>2</sup>	8.3

<sup>a</sup> All samples have 1700Å of Al, except sample #3 which has 940Å

<sup>1</sup> Fluence =  $5.15 \times 10^{20}$  atoms/cm<sup>2</sup> ( $\approx 0.5$  yr SSF)

<sup>2</sup> Fluence =  $1.15 \times 10^{21}$  atoms/cm<sup>2</sup> ( $\approx 1.1$  yr SSF)

<sup>3</sup> Fluence =  $8.99 \times 10^{20}$  atoms/cm<sup>2</sup> ( $\approx 0.8$  yr SSF)

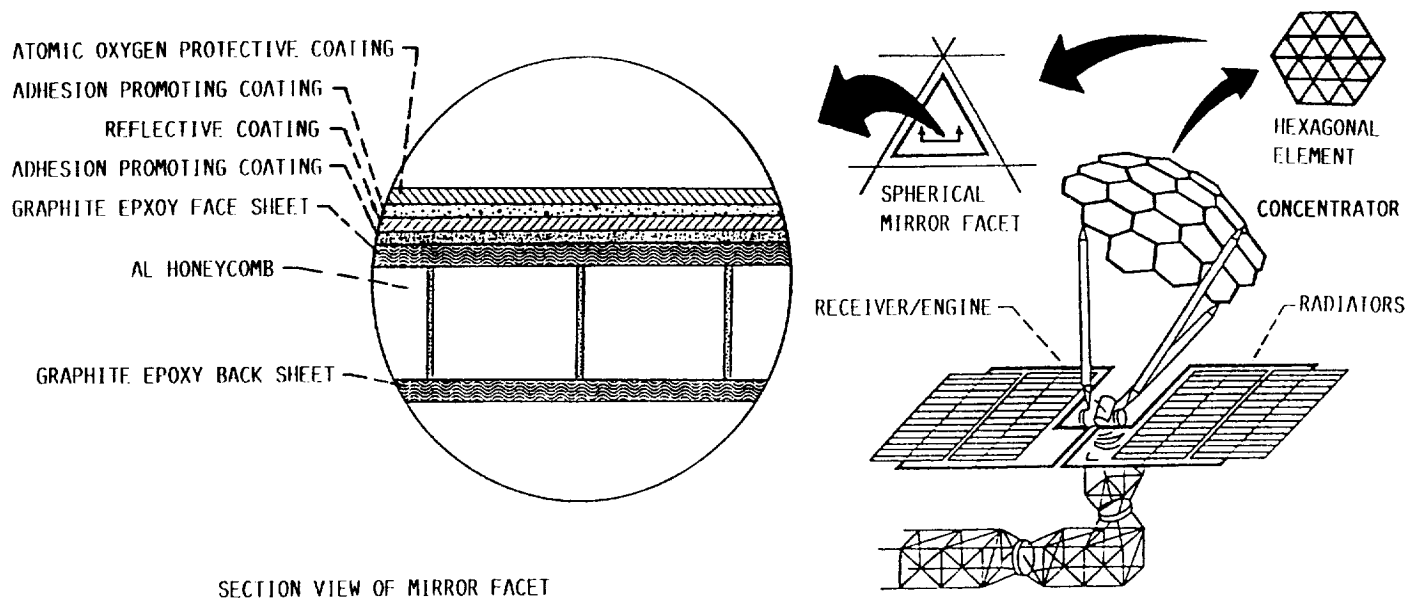


FIGURE 1. - SOLAR CONCENTRATOR CONFIGURATION FOR SPACE STATION FREEDOM SOLAR DYNAMIC POWER MODULE<sup>2</sup>.

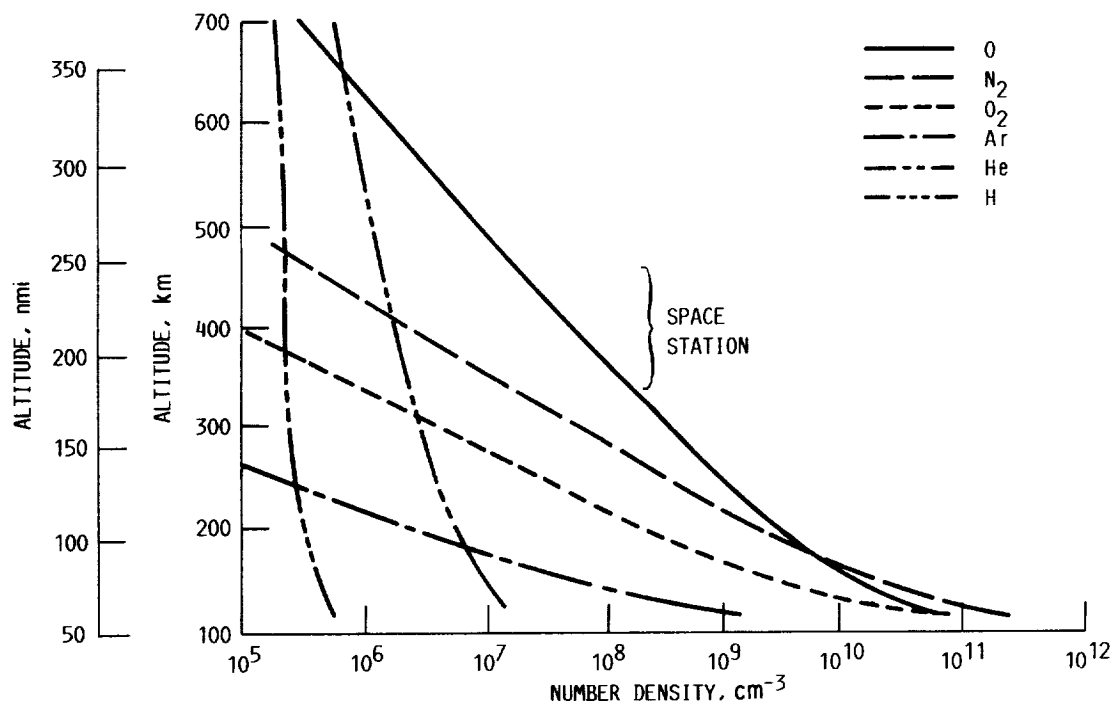
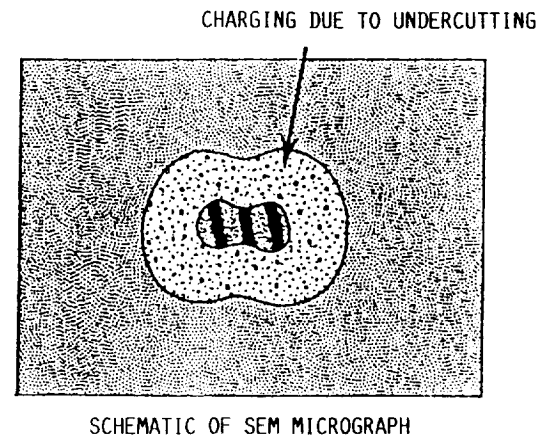
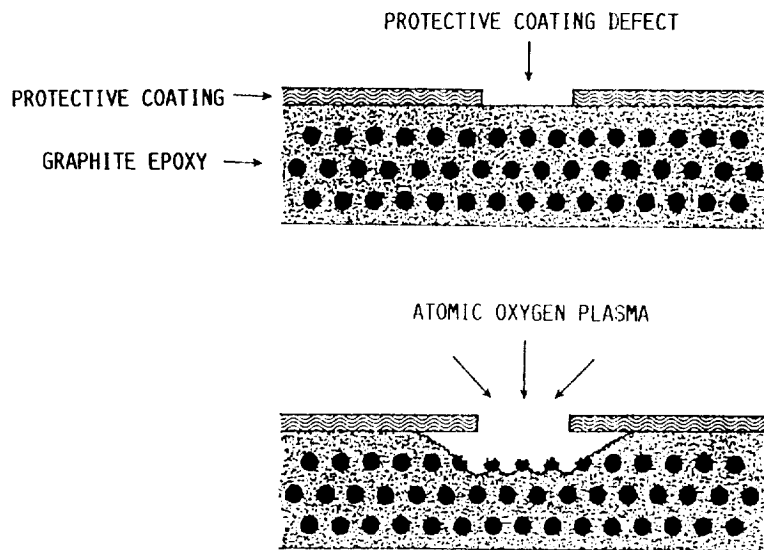
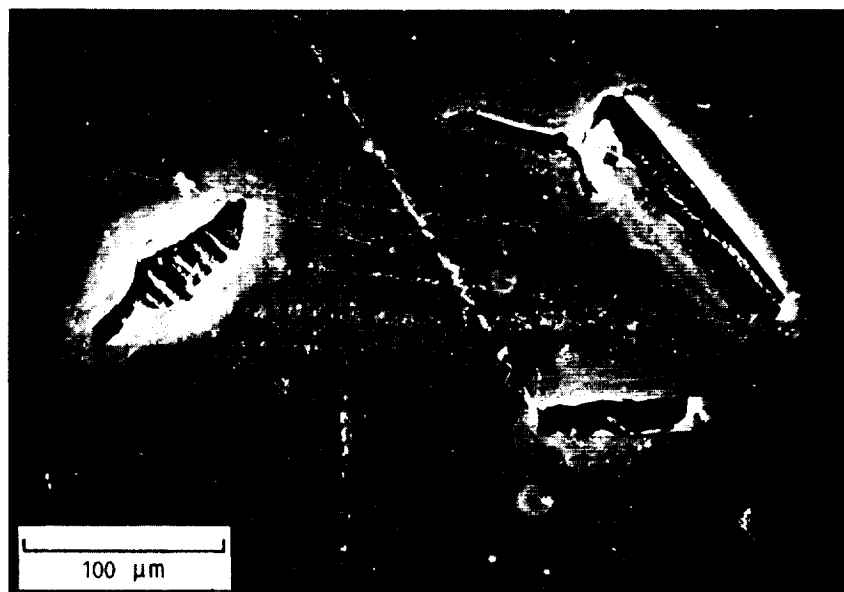


FIGURE 2. - ATMOSPHERIC COMPOSITION OF LEO AS A FUNCTION OF ALTITUDE<sup>5</sup>.



(a) ILLUSTRATION OF UNDERCUTTING IN AN ATOMIC OXYGEN PLASMA ENVIRONMENT.



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OF POOR QUALITY

(b) ATOMIC OXYGEN DAMAGE AFTER PLASMA ASHING TO AN EFFECTIVE FLUENCE OF  $1.15 \times 10^{21}$  ATOMS/ $\text{cm}^2$ . THIS SAMPLE HAS THE SAME PROTECTIVE, REFLECTIVE, AND SUBSTRATE MATERIALS AS CURRENTLY PLANNED FOR SSF CONCENTRATOR SURFACES.

FIGURE 3. - ATOMIC OXYGEN UNDERCUTTING AT DEFECT SITES IN PROTECTIVE COATINGS ON GRAPHITE EPOXY.

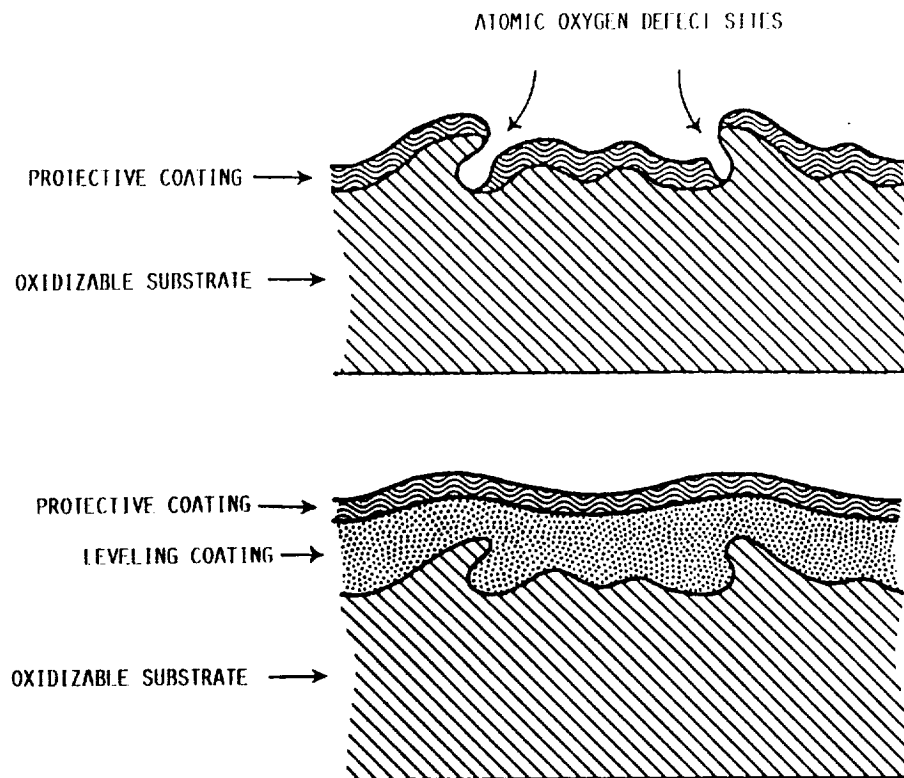


FIGURE 4. - LEVELING COATINGS PROVIDE INCREASED ATOMIC OXYGEN DURABILITY BY SMOOTHING ROUGH SUBSTRATE SURFACES, ALLOWING THE EVAPORATIVE DEPOSITION OF CONTINUOUS REFLECTIVE AND PROTECTIVE FILMS.

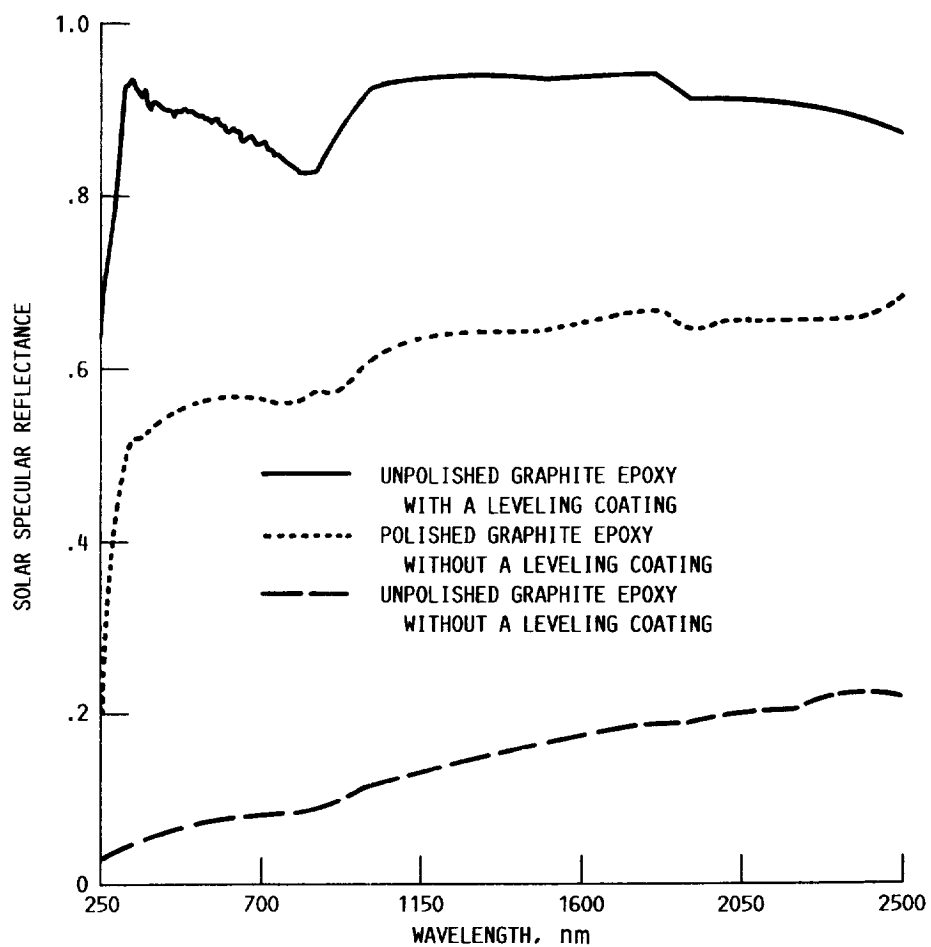
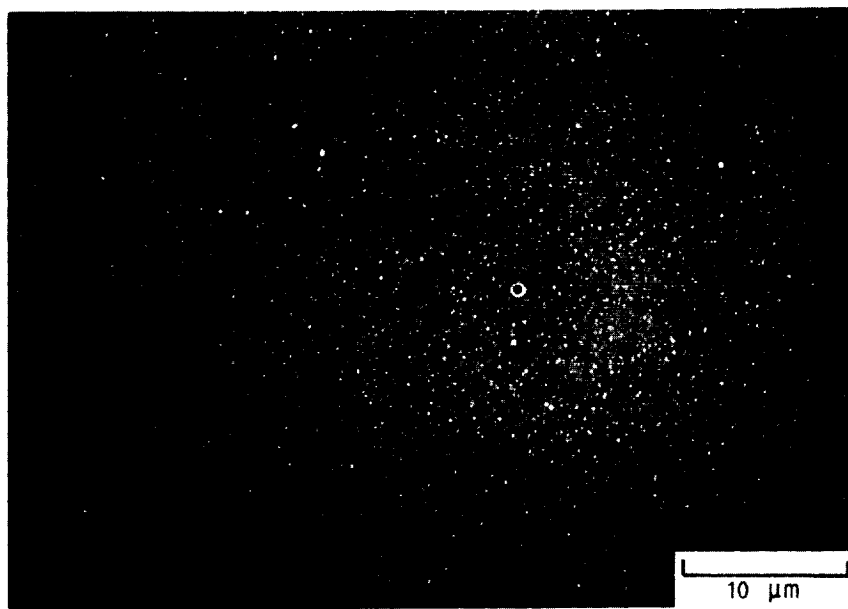
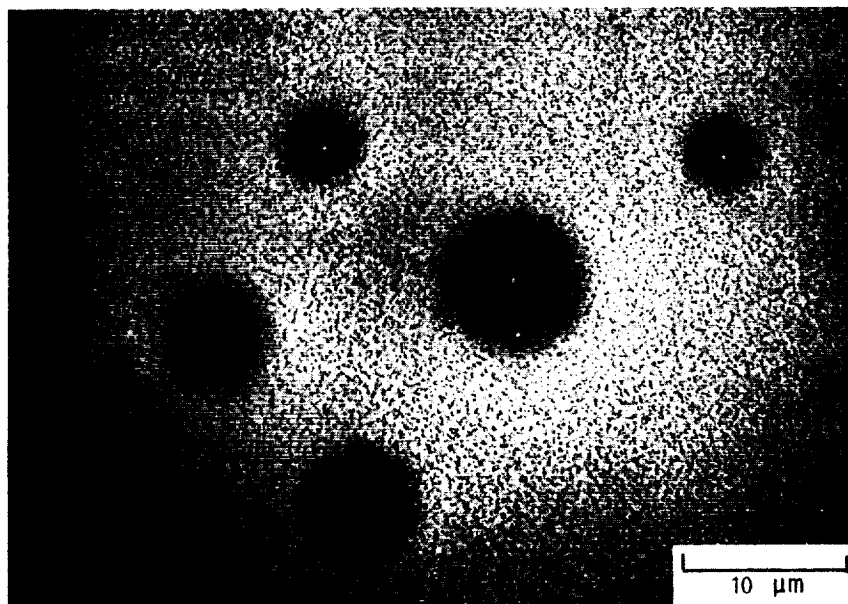


FIGURE 5. - INCREASE IN THE SOLAR SPECULAR REFLECTANCE OF Al-COATED GRAPHITE EPOXY WITH THE APPLICATION OF A LEVELING COATING.



(a) SECONDARY ELECTRON IMAGE (NORMAL SEM IMAGE). UNDERCUT AREAS ARE NOT VISIBLE.



(b) BACKSCATTERED ELECTRON IMAGE. UNDERCUT AREAS BECOME APPARENT AS DARK REGIONS AROUND SMALL DEFECTS.

FIGURE 6. - OBSERVATION OF UNDERCUTTING DURING SEM EXAMINATION OF A CONDUCTIVE SAMPLE.



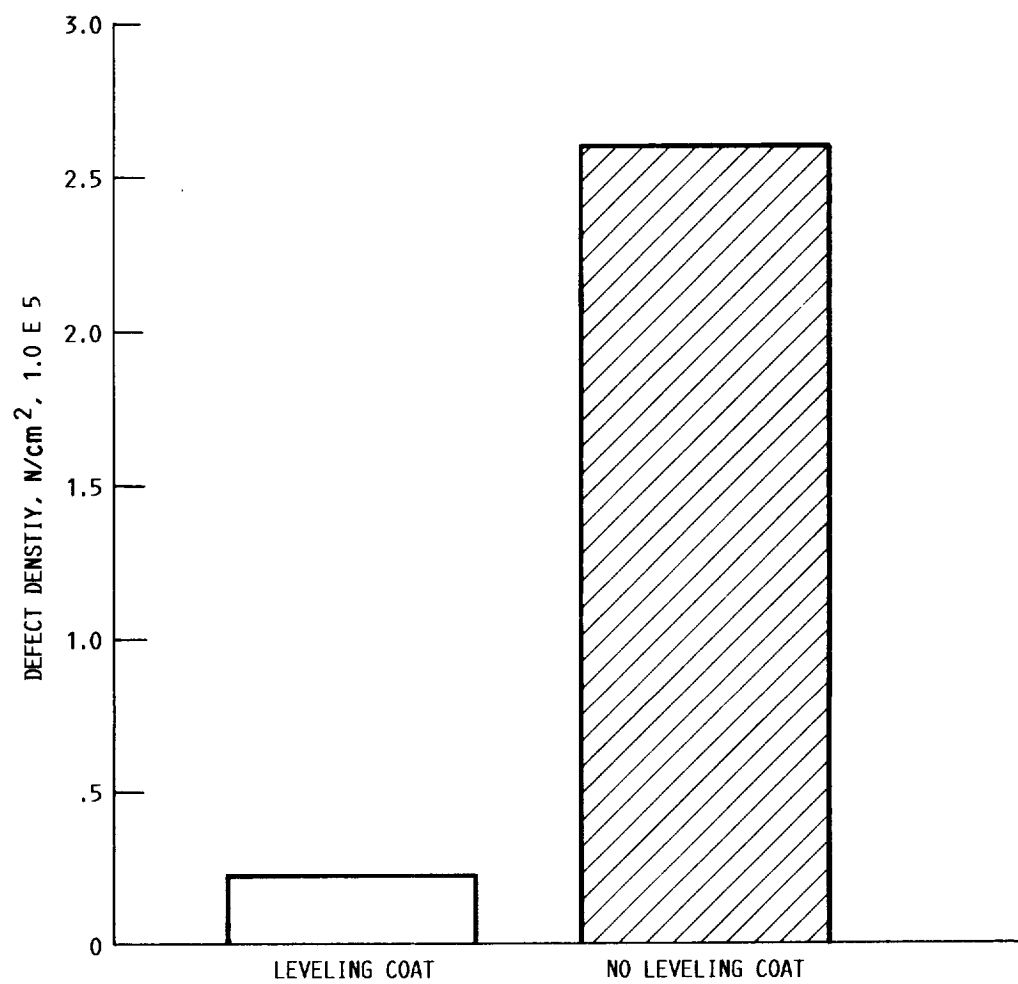
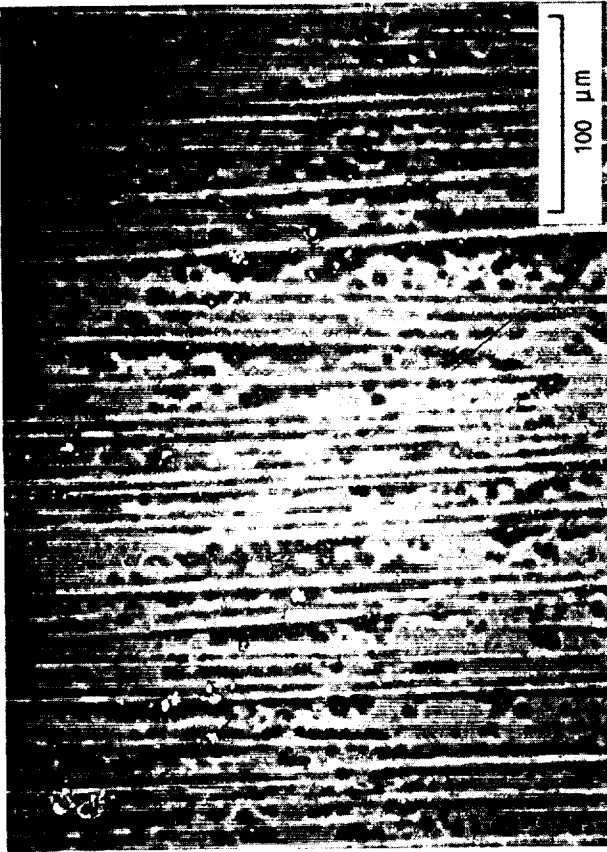
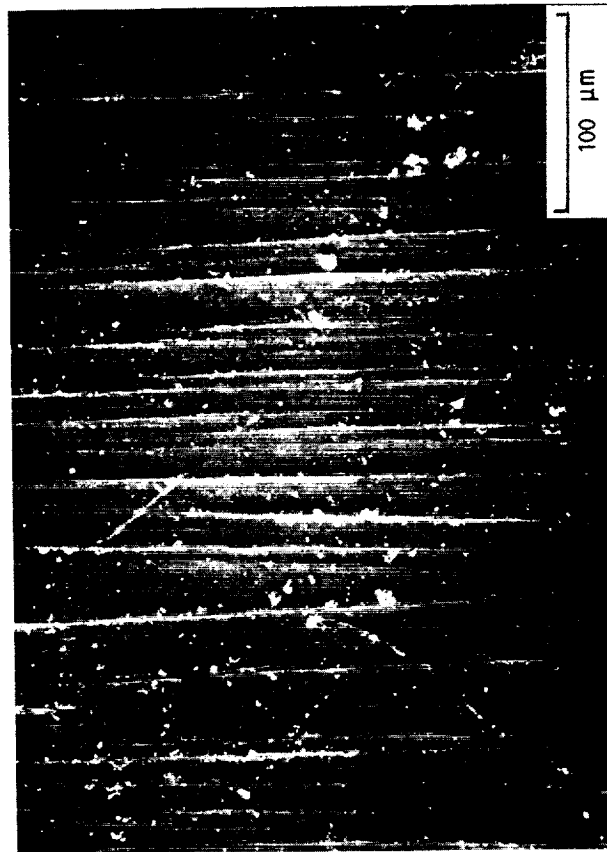


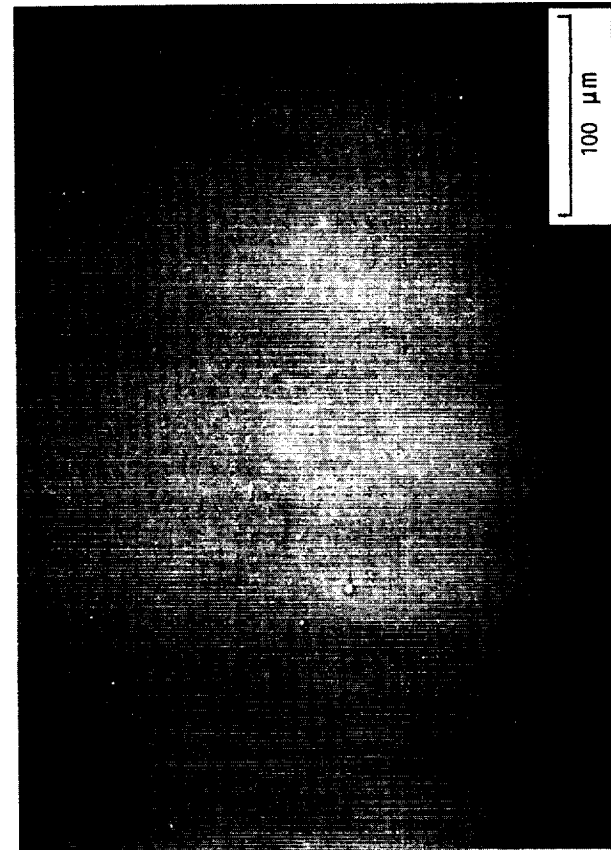
FIGURE 7. - ATOMIC OXYGEN DEFECT DENSITY OF Al-COATED GRAPHITE EPOXY WITH AND WITHOUT LEVELING COATINGS.



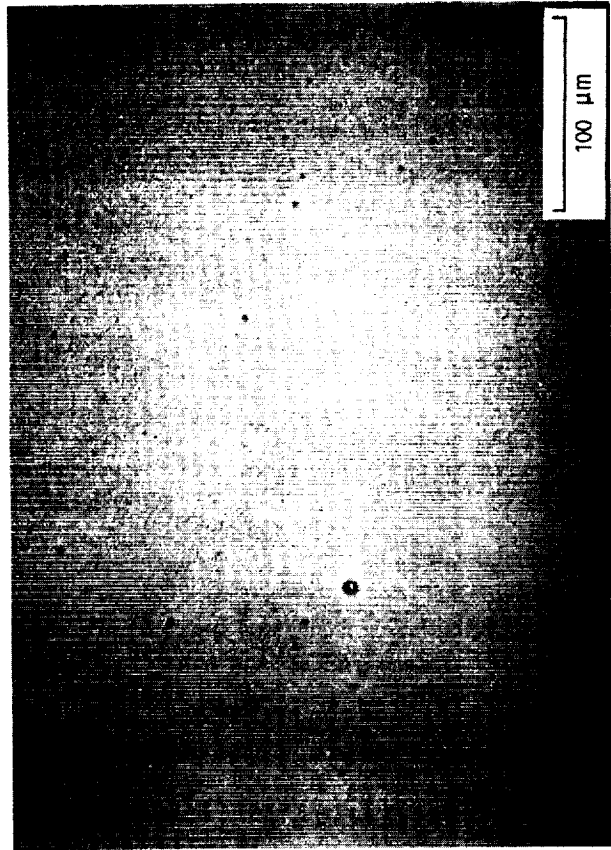
(b) BSE IMAGE OF 8a, DARK REGIONS REVEAL SEVERE UNDERCUTTING.



(a) SE IMAGE, SAMPLE WITHOUT A LEVELING COAT.



(c) SE IMAGE, SAMPLE WITH A LEVELING COAT.



(d) BSE IMAGE OF 8c, FEW UNDERCUT REGIONS ARE PRESENT.

FIGURE 8. - ATOMIC OXYGEN PLASMA ASHED Al-COATED GRAPHITE EPOXY WITH AND WITHOUT LEVELING COATINGS.

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7. Author(s) Kim K. de Groh, Therese M. Dever, and William F. Quinn				8. Performing Organization Report No. E-5375	
				10. Work Unit No. 506-41-31	
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16. Abstract Space power systems for Space Station Freedom will be exposed to the harsh environment of low Earth orbit (LEO). Neutral atomic oxygen is the major constituent in LEO and has the potential of severely reducing the efficiency of solar dynamic power systems through degradation of the concentrator surfaces. Several transparent dielectric thin films have been found to provide atomic oxygen protection, but atomic oxygen undercutting at inherent defect sites is still a threat to solar dynamic power system survivability. Leveling coatings smooth microscopically rough surfaces, thus eliminating potential defect sites prone to oxidation attack on concentrator surfaces. <b>This paper investigates</b> the ability of leveling coatings to improve the atomic oxygen durability of concentrator surfaces. The application of a EPO-TEK <sup>®</sup> 377 epoxy leveling coating on a graphite epoxy substrate resulted in an increase in solar specular reflectance, a decrease in the atomic oxygen defect density by an order of magnitude and a corresponding order of magnitude decrease in the percent loss of specular reflectance during atomic oxygen plasma ashing.					
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